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AIP Conference Proceedings -- April 1, 1991 -- Volume 219, Issue 1 pp. 510-517

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Effect of RF plasma deposition parameters on $Y_1Ba_2Cu_3O_{7-x}$ thin films

A. Shah, E. Narumi, J. Schutkeker, S. Patel, and D. T. Shaw
New York State Institute on Superconductivity, Amherst, NY 14260

Yttrium, barium and copper nitrates are dissolved in de-ionized water to form a 0.038M: 0.078M: 0.105M solution, which is used to generate, *in-situ* $Y_1Ba_2Cu_3O_{7-x}$ superconducting films in an argon-oxygen rf plasma. rf power is operated at 4.5 kW, 13.56 MHz and 760 Torr pressure. The best zero resistance temperature, onset critical temperature and current density measured by the four probe transport method are 86 K, 92 K and 4×10^5 A/cm² at 77 K and zero field. X-ray diffraction shows the films to be oriented with *c*-axis perpendicular to the substrate surface. Variations in the critical temperatures of the films and their microstructure depend on rf power, solution composition, solution concentration, plasma gas and aerosol carrier gas. It has been seen that the concentration of barium in the solution is more critical than that of copper. The concentration of the solution is related to the rf power, to prepare the best film, the concentration and the power levels have to be matched. While the variation of both aerosol carrier gas flow rate and the plasma gas flow rate causes a change in the structure and critical temperature of the film, the control of the former is more critical than the plasma gas flow rate.

PACS: 74.70.Jm, 81.15.Gh, 74.75.+t [Additional Information](#)

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**Applied Physics Letters -- November 2, 1992 -- Volume 61, Issue 18 pp.
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Device-quality copper using chemical vapor deposition of β -diketonate source precursors in liquid solution

Bo Zheng, Eric T. Eisenbraun, Jun Liu, and Alain E. Kaloyeros

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(Received 27 May 1992; accepted 20 August 1992).

Device-quality copper films were produced by chemical vapor deposition from copper(II) β -diketonate precursors using a liquid delivery approach. This liquid delivery method exploits the high solubility of copper(II) β -diketonate precursors in selected solvents, such as isopropanol and ethanol, to provide highly accurate, reproducible, and controllable flow rates of precursor and solvent mixtures to the reaction zone. The approach was successfully employed to produce high-quality copper films from predetermined mixtures of bis(hexafluoroacetylacetonato) copper(II) and ethanol or isopropanol. Plasma-assisted chemical vapor deposition (PA-CVD) was used with substrate temperatures of 160–170 °C, reactor working pressures of 1.0–1.7 Torr, hydrogen flow rates between 500 and 1200 cc/min, and hydrogen plasma power density ranging from 0.13 to 0.25 W/cm². The films were subsequently characterized by Rutherford backscattering spectroscopy, cross-section SEM (scanning electron microscopy), and a four-point resistivity probe. These studies indicated that the films thus grown were pure, dense, highly uniform, and displayed resistivities of 1.7–1.9 $\mu\Omega$ cm. Films produced on patterned test structures exhibited conformal step coverage and complete hole and via fill. Growth rates over large-area substrates were around 250 Å/min for via filling. Applied Physics Letters is copyrighted by The American Institute of Physics.

DOI: 10.1063/1.108286

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Hong Jie Jin *et al.*, [J. Vac. Sci. Technol. A](#) **17**, 726 (1999)
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**Journal of Materials Research -- November 1990 -- Volume 5, Issue 11 pp.
2387-2397**

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CVD diamond deposition processes investigation: CARS diagnostics/modeling

Stephen O. Hay, Ward C. Roman, and Meredith B. Colket III
United Technologies Research Center, East Hartford, Connecticut 06108,

The driving force behind the strong interest in diamond deposition processes is the outstanding combination of unique natural properties of this material. A wide variety of techniques has been employed to generate diamond coatings including hot filament, thermal plasma, CVD, PACVD (rf, dc, and microwave), low energy carbon ion beam, laser beam, oxyacetylene torch, and numerous hybrid dual-beam configurations. Thus, there are many routes available for producing diamond coatings in the form of small individual crystals, amorphous coatings, polycrystalline films or single crystal films under conditions far removed from the thermodynamically stable region nominally associated with diamond growth. CVD of diamond coatings from hydrocarbon containing gases can have an almost infinite number of compositions and structures; each with differing amounts of sp^3 (diamond) and sp^2 (graphite) bonding. This variation has contributed to confusion both in the working definition of diamond coatings and in understanding the controlling processes of forming these films. In fact, the mechanisms involved in the gas phase processes, the nucleation and growth structures, and especially their correlation are poorly understood. Interestingly, this limitation has not hindered the headstrong rush for commercial development. Commercial deployment of this nascent technology hinges on the ability to form uniform coatings at high deposition rates over large and irregular surface areas. Intrinsic to the ability to scale existing techniques for required deposition rates over large areas is detailed mechanistic knowledge of both the gas phase and gas surface reactions which result in high purity deposition of the proper morphology. In the case of diamond deposition, the focus of most studies has been to document the effect of

varying macroscopic physical observables (input energy, flow rates, total pressures, etc.) upon coating characteristics such as purity, morphology, hardness, and adhesion. Chemical kinetic modeling has concentrated on boundary conditions. Models generally contain detailed knowledge of both initial conditions and the final state but have an incomplete picture of the transitional process. To address this dearth of experimental data, a variety of remote, nonintrusive optical techniques have been applied to probe the species, concentrations and energetics of these transitional regions. We have applied coherent anti-Stokes Raman spectroscopy (CARS), using a narrowband, scanned colinear configuration to measure temperatures, relative concentrations and detect species in low pressure CVD of polycrystalline diamond. CARS measurements were obtained for methane, hydrogen and acetylene in either or both a rf plasma reactor and a hot filament reactor. In the rf PACVD experiments a mixture of 1 CH₄ in H₂ was used at a total pressure of 5 Torr. The rf power input to the plasma was 300 watts and the H₂ and CH₄ flow rates were 99 and 1 sccm, respectively. As acetylene (C₂H₂) has been proposed as an intermediate in diamond growth, it was selected for the initial series of measurements. In the absence of rf power, a sensitivity of 5 mTorr was observed; in the plasma downstream of the rf coils, no observable signal attributable to C₂H₂ was evident. This places an upper limit to conversion of methane to acetylene at 20, a figure representing the observed sensitivity to C₂H₂. In the hot filament reactor, the gas flow was 200 sccm of 1 CH₄ in H₂ at a total pressure of 150 Torr. Under these conditions, C₂H₂ was detectable. Absolute concentrations were not calculated but the observed spectra is within an order of magnitude of our sensitivity limit. This allows estimation of the C₂H₂ partial pressure near the substrate as 5–50 mTorr or from 0.66 to 6.6 conversion from methane. In view of this low conversion percentage the absence of a signal in the rf experiments must be taken as inconclusive. CARS spectra of methane were also obtained in both reactors. In the rf reactor, under similar conditions to those described previously, the methane relative concentration decreased to 25 as the rf power was increased from zero to 400 watts. In the hot filament reactor, CH₄ CARS signal profiles were obtained as a function of axial distance from the hot filament, and parametrically as a function of filament temperature. Comparison of these profiles, in which the observed signal decayed monotonically as the filament was approached and increased monotonically downstream of the filament was made with theoretical calculations. This comparison showed that the fluctuations were attributable to temperature/pressure effects and not to chemistry. In an effort to determine if the observed depletion in the rf plasma was similarly attributable, the CARS signal of hydrogen was observed as a function of axial distance downstream of the rf coil centerline and parametrically as a function of rf power. In contrast to expected behavior in the thermal hot filament reactor, little rotational excitation was observed in the plasma. Rotational temperatures were assigned to hydrogen based upon comparison with theoretically derived spectra. At 450 watts of rf power, rotational temperatures of 340 K were observed 4 to 6 cm downstream of the coil, the region where the 25 decrease in CH₄ was observed. Little or no density fluctuations accrue due to these temperatures, indicating that the observed depletion in methane signal is attributable to

decomposition or chemical reaction in the plasma. In summary, CARS is applicable to reactant species (CH_4) axial profiling in both reactors, but can be limited by sensitivity in the detection of intermediate or product species (C_2H_2). In addition, CARS thermometry can be utilized to profile the rotational temperatures of selected species.

PACS: 81.15.Gh, 42.65.Dr [Additional Information](#)

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